

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**



APPLICANT(S) : John Kollar

EXAMINER :

SERIAL NO. : 08/567,564

ART UNIT :

FILING DATE : December 5, 1995

ASSIGNOR : Redox Technologies  
Inc.

TITLE : PREPARATION OF DIALKYL PEROXIDES

**DECLARATION OF JOHN CHRISTOPHER KOLLAR D.O.**

I, JOHN CHRISTOPHER KOLLAR D.O., the undersigned, depose and say  
that:

1. I am a surgeon, having obtained a B.S. degree from Rutgers University in 1982, and a D.O. degree from UMDNJ, the University of Medicine and Dentistry of New Jersey in 1987. I have also completed my internship and surgical residency at Saddle Brook Hospital in 1991. I have taken advanced placement chemistry in high school and was appointed a Rutgers Scholar in chemistry, doing advanced research on potential carcinogenic antagonists. From the summer of 1977 until entering medical school in late summer of 1982, I worked summers, holiday periods and some weekends, as my college schedule permitted, as a chemical technician for Redox Technologies Inc. I worked in many areas of novel research with great commercial potential. As part of my work, I understood the chemical, economical and practical aspects of my efforts. I worked extensively in oxidations, peroxidations, epoxidations, dimerizations, free radical induced

coupling, alkylations, esterifications, separations, a variety of catalysis reactions including the use of acidic and basic resins, analysis of various kinds of process systems, from batch to flow systems, from vapor phase to liquid phase systems, at low to high pressure.

2. I reacted *iso*-butylene with *t*BHP (*tertiary* butyl hydroperoxide) in the presence of an effective amount of a substantially solid, insoluble, heterogenous acidic catalyst on June 29, 1979, as evidenced by the copy of my laboratory notebook entry attached hereto as Exhibit "1-a". These reactions were conducted by myself at the direction of my father, John Kollar, for the purpose of developing a commercially economical process for the production of DtBP (*di-tertiary* butyl peroxide), which was a necessary reactant for a methanol-based EG (ethylene glycol) process on which I was also working under my father's direction.

3. I carried out three experiments. In the first two experiments, *iso*-butylene, which I abbreviated as "*iso*-C<sub>4</sub>" in my laboratory notebook entry, Exhibit "1-a", was reacted with methanol, which I abbreviated as "Sol'n 1", in the presence of "Resins", which was my abbreviation for Amberlyst A-15, a strong acid ion exchange resin of the sulfonic acid type. In both experiments, 2 g of the acidic catalyst were employed; 10 g of "Sol'n 1" was used, which as Exhibit "1-a" indicates, was made up from 22.5 g of toluene and 2.5 g of methanol, which I abbreviated as "MeOH"; and the reactions were carried out for 1 hour at about 60° C. Twice as much of the *iso*-butylene was used in the second experiment as in the first. Both of these experiments were controls, in that they utilized all of the components and conditions of the reaction except for the *tert*-butyl hydroperoxide reactant. As is noted in Exhibit "1-a", gas chromatographic analysis indicated a yield of methyl-*tert*-butyl ether, abbreviated as "MtBE", of about 3.7% and about

5.6%, for each experiment, respectively. The entries in my laboratory notebook relating to the results of the gas chromatographic analysis were made shortly after the analyses were completed by my father, John Kollar, who had carried out the analyses on the reaction products shortly after the reactions were completed. He also verbally communicated those results to me at that time.

4. In the third experiment, I reacted 1.5 g of *tert*-butyl hydroperoxide, which I abbreviated "*t*-butyl hydro" in my laboratory notebook entry Exhibit "1-a", with 0.95 g of *iso*-butylene, with all of the other conditions being the same as in the first two experiments. The reaction successfully produced di-*tert*-butyl peroxide in a 78% yield, as recorded in Exhibit "1-a" by my father, John Kollar in accordance with the circumstances described in the paragraph above. The notebook entry indicates a weight, peroxide titer and calculation for the amount of *tert*-butyl hydroperoxide reacted to form di-*tert*-butyl peroxide. Thus, I received confirmation of the positive results for the reactions which occurred on June 29, 1979, shortly thereafter when my father, John Kollar, verbally communicated those positive results to me and entered them on the same page of my laboratory notebook that contained the record of the original reactions, as evidenced by the attached laboratory notebook entry Exhibit "1-a".

5. All statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further, these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the above-identified application or any patent issued thereon.

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DATE

John Christopher Kollar  
JOHN CHRISTOPHER KOLLAR